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IS 1448-114 (1984): Methods of test for petroleum and its products, Part 114: Determination of congealing point of petroleum waxes [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 114]

DETERMINATION OF CONGEALING POINT OF PETROLEUM WAXES

UDC 665·6/·7 : 665·772 : 536·421·4

Adapted with permission from the Joint Publication ASTM Designation D 938-71 (Reapproved 1981)/IP 76/70 (Reapproved 1978) and ISO 2207-1980

1. SCOPE

1.1 This method covers the test for determining the congealing point of petroleum waxes, including petrolatum.

2. OUTLINE OF THE METHOD

2.1 A sample of wax is melted and a droplet is made to adhere to the bulb of a thermometer. Using a prewarmed flask as an air jacket, the droplet on the bulb is allowed to cool at a fixed rate until it congeals. The congealing point is observed as the temperature at which the droplet ceases to flow as the thermometer is turned.

3. SIGNIFICANCE

3.1 Congealing point is a wax property that is of interest to many petroleum wax consumers. The procedure described here measures the temperature at which a sample being cooled develops a ' set ' or resistance to flow. At that temperature, the wax may be at or close to the solid state, or it may be semisolid and quite unctuous, depending on the composition of the wax or petrolatum being tested. In the case of petrolatums, congealing property is associated with the formation of a gel structure as the sample cools.

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4. DEFINITION

4.1 Congealing Point — It is the temperature at which molten petroleum wax, when allowed to cool under prescribed conditions, ceases to flow.

5. APPARATUS

5.1 Thermometer* — Conforming to the following requirements:

Range	+ 20 to + 100°C
Immersion	Total
Graduation at each	0·2°C
Longer lines at each	1°C
Figured at each	2°C
Maximum line width	0·15 mm
Scale error not to exceed	0·2°C
Expansion chamber	110°C
Permitting heating to overall length	305 to 315 mm
Stem diameter	6·0 to 8·0 mm
Bulb shape top finish	Ellipsoidal
Bulb length	10 to 12 mm
Bulb diameter	4·5 to 6·0 mm
Distance from bottom of bulb to 20°C line	60 to 70 mm
Length of graduated portion	170 to 215 mm
Top finish	Ring

5.2 Erlenmeyer Flask — 125 ml, glass, to serve as a thermometer jacket.

5.3 Cork or Rubber Stopper — For attaching the thermometer to the Erlenmeyer flask.

6. PROCEDURE

6.1 Adjust the thermometer through the stopper so that the bottom of the bulb will be 10 to 15 mm above the bottom of the Erlenmeyer flask when the stopper is fitted snugly in the flask. After making this adjustment, remove the thermometer and stopper from the flask, being careful not to change the position of the stopper relative to the thermometer stem.

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*Thermometers ASTM 54C and IP 18C conform to these requirements.

6.2 Place approximately 50 g of sample, which is representative of the material under inspection, in a porcelain evaporating dish or other suitable container.

6.3 Place the empty Erlenmeyer flask (without the thermometer assembly) and the container holding the sample in a temperature-controlled oven set at 99°C ± 3°C until the sample and flask reach oven temperature (*see* Note).

NOTE — For nonreferee, routine testing of samples known to have low congealing points, the oven may be set at a lower temperature, but it shall be at least 11°C above the expected congealing point of the sample.

6.4 Remove the sample from the oven and completely immerse the thermometer bulb in it without covering any part of the thermometer stem with sample. Gently stir the sample with the thermometer until the mercury column has stopped rising.

6.5 While holding the thermometer bulb in the molten wax sample, remove the heated flask from the oven, using a towel or gloves to protect the hands. Now carefully remove the thermometer from the sample, taking care to retain a relatively large drop of sample adhering to the bulb. Holding the thermometer in a horizontal position, firmly fit the thermometer and stopper into the flask. Keep the assembly in a horizontal position.

6.6 While observing the drop on the thermometer bulb at an eye level position, rotate the thermometer and flask about a horizontal axis. Use a steady and even rate for each continuous full revolution, and complete each revolution in not less than 2 seconds, nor more than 3 seconds. Do not pause at the completion of each revolution any longer than required to reindex the fingers for the next full and continuous rotation (*see* Note). When the drop is observed to rotate with the bulb, immediately read the thermometer to the nearest 0·25°C and record this determination. Make a repeat determination on the wax sample. If the variation of these two determinations does not exceed 1°C, record the average of these determinations as the congealing point of the sample under test. If the variation of two determinations is greater than 1°C, make one additional determination and record the average of the three determinations as the congealing point.

NOTE — Operators should periodically check themselves for compliance with this turning rate. The brief pause time is not to be included in the 2 to 3 seconds rotation.

7. REPORT

7.1 Report the average of the multiple determinations as the congealing point, shown to the nearest 0·25°C.

8. PRECISION

8.1 Results of duplicate tests shall not differ by more than the following amounts:

	Repeatability	*Reproducibility*
Distillate waxes	0·5°C	1·5°C
Residual waxes, including petrolatums	1·0°C	2·5°C

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